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Citation: APL Materials **2**, 116101 (2014); doi: 10.1063/1.4900816
View online: http://dx.doi.org/10.1063/1.4900816
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APL MATERIALS **2**, 116101 (2014)

Low temperature photoresponse of monolayer tungsten disulphide

Bingchen Cao,[1] Xiaonan Shen,[1] Jingzhi Shang,[1] Chunxiao Cong,[1] Weihuang Yang,[1] Mustafa Eginligil,[1,a] and Ting Yu[1,2,3,a]
[1]*Division of Physics and Applied Physics, School of Physical and Mathematical Sciences, Nanyang Technological University, Singapore, 637371*
[2]*Department of Physics, Faculty of Science, National University of Singapore, Singapore, 117542*
[3]*Graphene Research Centre, National University of Singapore, Singapore, 117546*

(Received 29 July 2014; accepted 20 October 2014; published online 3 November 2014)

High photoresponse can be achieved in monolayers of transition metal dichalcogenides. However, the response times are inconveniently limited by defects. Here, we report low temperature photoresponse of monolayer tungsten disulphide prepared by exfoliation and chemical vapour deposition (CVD) method. The exfoliated device exhibits n-type behaviour; while the CVD device exhibits intrinsic behaviour. In off state, the CVD device has four times larger ratio of photoresponse for laser on/off and photoresponse decay–rise times are 0.1 s (limited by our setup), while the exfoliated device has few seconds. These findings are discussed in terms of charge trapping and localization. © *2014 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License.* [http://dx.doi.org/10.1063/1.4900816]

The family of transition metal dichalcogenides (TMDs) has members which have many technologically important properties and physically interesting states.[1] When they are thinned to few layers and become 2D materials,[2] they may present new phases of charge density waves[3] and superconductors,[4] or semiconductors.[5] Among these, 2D semiconductor TMDs, as well as their heterostuctures[6] and hybrid structures,[7] are promising for a variety of potential optoelectronic applications owing to their direct band gap at K valley in monolayers.[8] Some applications can be named as high on and off ratio phototransistors and fast photodetectors,[9] as well as future devices based on spin-valley coupling.[10] In order to make standardized, effective, and dynamic optoelectronic devices based on TMDs, there are three major issues to elaborate: (i) to produce single crystal, large area samples with strong homogeneous luminescence, (ii) to fabricate devices with high but tunable photoresponse, and (iii) to find the right TMDs for rapid yet well-defined response and detection reflecting intrinsic semiconducting properties.

There has been important advance in dealing with the first issue by mastering on growth techniques. Over the last few years, strong excitonic photoluminescence of monolayer TMDs, MoS_2, and WS_2 has been demonstrated, particularly in mechanically exfoliated single crystal flakes with sizes less than 10 μm.[11] Interesting strain[12] and thermal conductance[13] properties were observed in these exfoliated samples. Growth of large-area, while maintaining similar high quality, flakes of MoS_2 and WS_2 on SiO_2 and WS_2 on sapphire substrates, was succeeded by chemical vapour deposition (CVD) methods.[14–16] Recently by utilizing an advanced CVD method, triangular flakes of WS_2 with size exceeding 250 μm were grown.[17] By this method, it is possible to obtain monolayer WS_2 flakes with homogeneous, high crystalline quality, and nonblinking luminescence[18] which are stronger than their exfoliated counterparts and their cousin MoS_2. Tunable emission by electrical gating[19] and chemical doping[20] has been demonstrated for these CVD grown flakes.

[a]Authors to whom correspondence should be addressed. Electronic addresses: yuting@ntu.edu.sg and meginligil@ntu.edu.sg

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Solutions to the issues related to optoelectronic device performance have been guided by the extensive knowledge of graphene, which is the leading 2D material without a natural band gap. High photoresponsivity (8.61 A/W) of graphene was reported,[21] when graphene is lithographically designed into graphene quantum dots, which is about three orders of magnitude higher than that of graphene photoresponse reported previously.[22] The main reason for this was defects in the mid gap band. However, control over defects as well as the origin of the photocurrent is not well understood.[21] Instead of artificially created gaps and such structures it would be more beneficial to use natural semiconductors, such as 2D TMDs which could be precisely controlled by tuning the device both electrically and optically. Recently, a phototransistor based on monolayer MoS_2 in off state and with sub-micro watt illumination intensity showed a photoresponse of 880 A/W.[23] A photoresponse of four orders of magnitude higher than that of MoS_2 phototransistors was reported for graphene/MoS_2 heterosturctures.[24] However, in both cases, charge trapping either in MoS_2 or in the interface of MoS_2 and the substrate cause to slow down the response.

Although ultragain photoresponse has been obtained in 2D heterosturctures, one of the most important components of future's technology, fast and reliable photodiodes require high speed response and detection. Since trapped charges, which are mainly due to natural defects in MoS_2, are the reason for slow carrier recombination,[24] alternative 2D TMDs such as WS_2 and WSe_2 which are relatively more intrinsic and less defective[25] have attracted attention. In order to investigate TMD device photoresponse independent of the interface effects, low temperature characterizations are needed in which phonon assisted processes are expected to be negligible.[26] Although, a field effect transistor (FET) device based on 20 nm thick mechanically exfoliated WS_2 with photoresponsivity of 0.86 A/W,[27] a photosensor based on few layer WS_2,[28] and electrical measurements of monolayer WS_2 FET at room temperature[16,29] have been reported; there are no photoresponse of monolayer WS_2 FET studies up to date.

In this work, we compare and contrast low temperature photoresponse of two types of monolayer WS_2 FET devices, one grown by CVD and the other obtained by mechanical exfoliation. The exfoliated FET device exhibits n-type behaviour, large dark current, and low tunability in off state by laser illumination; while the CVD FET device exhibits intrinsic semiconducting behaviour, small dark current, and high tunability in off state by laser illumination. Further, time-resolved measurements *reinforce* the argument of the earlier optical measurements[18] of CVD grown WS_2 about having relatively *less defects*, and *underline* the physics behind the response, and the detection *timescales* which are vital for optoelectronic applications, such as photodiodes.

Monolayer WS_2 flake was mechanically exfoliated from bulk crystal onto Si substrate with 300 nm SiO_2; then processed into a device labelled as S1-EXF, by e-beam lithography with Ti (5 nm)/Au (75 nm) contacts for two probe measurements. The other sample is a triangular monolayer flake which is grown by a unique CVD method, as described elsewhere.[17] Similarly, the CVD grown triangular flake was picked among intense, homogeneous, high crystalline quality flakes,[18] and processed into device S2-CVD. Micro-photoluminescence (PL) of S1-EXF and S2-CVD, by 532 nm laser excitation at room temperature, in Figs. 1(a) and 1(b), respectively, demonstrates the quality of the flakes after device fabrication. There is single neutral exciton peak at 1.965 eV in intrinsic S2-CVD. However, as a result of n-type doping, there is a blueshift in the neutral exciton peak in S1-EXF of 50 meV (2.015 eV). Also there is an additional peak at 1.98 eV, which is attributed to charged exciton with relatively lower PL intensity.[18] In the upper part of the insets to Figs. 1(a) and 1(b), the optical image of S1-EXF (S2-CVD) is shown. Bright fluorescence images corresponding to the optical images, in the lower parts of the insets to Figs. 1(a) and 1(b), are in agreement with the PL data for S1-EXF and S2-CVD.

Initial electrical measurements of these FET devices were performed at room temperature and 4.2 K, at dark. At zero gate voltage, drain current vs. drain voltage of S1-EXF and S2-CVD are compared in Fig. 1(c) up to 5 V. Both devices show similar behavior up to 2 V, but current is about twice at 5 V in S1-EXF. This voltage was sufficient to observe the device activation in S1-EXF without device heating. When the temperature is lowered to 4.2 K, 10 nA of current at room temperature at 5 V drain voltage in S1-EXF improved to 350 nA by overcoming the Schottky barrier at ~2.5 V (Fig. 1(d)), while there is almost constant current of less than 1 nA in S2-CVD. Higher conductance at lower temperatures is typical for a heavily doped semiconductor,[30] which is the case

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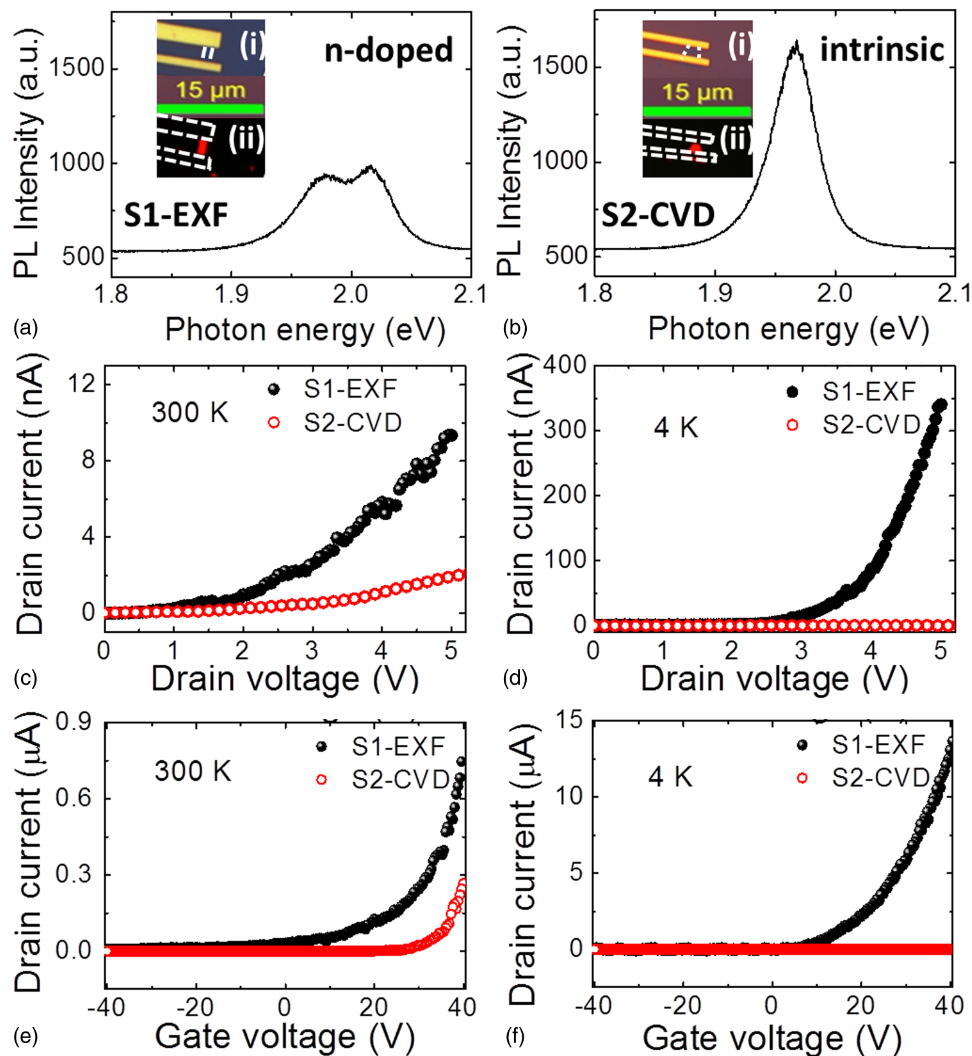

FIG. 1. PL spectra for (a) exfoliated sample S1 and (b) CVD sample S2 for 532 nm laser excitation. Insets show the corresponding optical and fluorescence images of the devices. Drain current vs. drain voltage for S1-EXF and S2-CVD (c) at room temperature (d) 4.2 K are shown. Gate voltage dependence of drain current for S1-EXF at 5 V drain voltage and S2-CVD at 16 V drain voltage (e) at room temperature (f) at 4.2 K are shown. Results suggest S1-EXF n-type doped, while S2-CVD intrinsic.

for S1-EXF. On the other hand, an intrinsic and thermally activated semiconductor is expected to have lower conductance at lower temperatures, as observed by the change from an activated S2-EXF in room temperature to deactivation at 4.2 K. Furthermore, gate voltage is varied from −40 V to 40 V, for 5 V (16 V) applied drain voltage for S1-EXF (S2-CVD). According to room temperature data shown in Fig. 1(e), S1-EXF FET needs 25 V gate voltage to overcome the threshold, while S2-CVD FET can turn on at about 0 V. At 4.2 K (Fig. 1(f)), the threshold drops below 0 V gate voltage in S1-EXF FET, as an indication of the Fermi level moving into the conduction band when temperature is lowered; while S2-CVD FET is off for the all applied gate voltages at 4.2 K, which is due to the suppression of thermally activated processes at low temperatures. The overall picture of high and low temperature drain current vs. drain voltage for geometrically similar devices, as well as drain current vs. gate voltage behaviour, is consistent for the argument of S1-EXF being n-type doped and S2-CVD being more intrinsic.

In order to investigate the response of these two samples with different electrical transport properties, the rest of the measurements were performed at 4.2 K, in the absence and existence of

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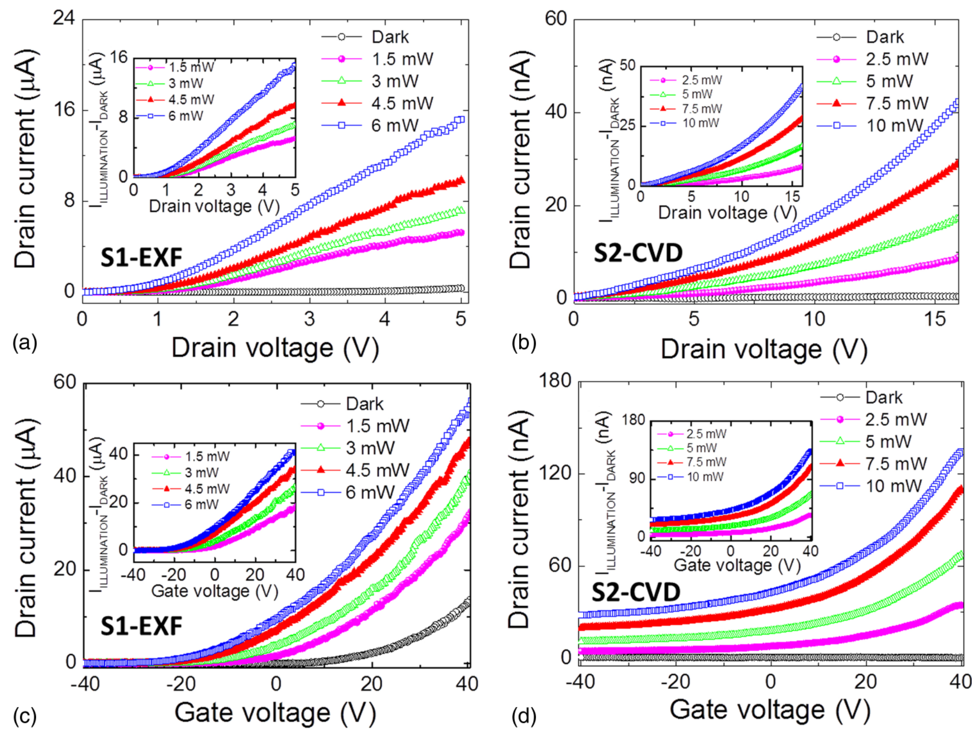

FIG. 2. At 4.2 K, drain current vs. drain voltage at dark, and under laser illumination with various powers are shown in (a) for S1-EXF and (b) for S2-CVD. Gate voltage dependence of drain current are shown in (c) at 5 V drain voltage for S1-EXF and (d) at 16 V drain voltage for S2-CVD. The insets plot the drain current due to illumination which is obtained by subtracting drain current data at corresponding illumination power ($I_{ILLUMINATION}$) from the drain current data at dark (I_{DARK}).

532 nm laser excitation. In Fig. 2(a), for S1-EXF, drain current vs. drain voltage characteristics at zero gate voltage reveal a nonlinear increase of the current with increasing laser powers for drain voltages over the Schottky potential. This nonlinear dependence in S1-EXF may be due to light doping as in the case of CdSe nanobelts,[31] and recombinations taking place in defect centers and traps.[26] On the other hand, the intrinsic sample S2-CVD exhibits a linear increase of the current with increasing laser powers (Fig. 2(b)). The nonlinear dependence is more noticeable when the Fermi level is increased by gating in S1-EXF, as seen in Fig. 2(c); whereas gating does not lead to any obvious change on linear dependence of photoresponse for device S2-CVD (Fig. 2(d)). When the gate voltage is lowered down to −40 V, there is a linear dependence of photoresponse with increasing laser power for both samples. This distinction between the photoresponse behaviour of S1-EXF and S2-CVD necessitates temporal measurements.

In off state, at −40 V gate voltage, and with highest drain voltage of 5 V (16 V), photoresponse of S1-EXF (S2-CVD) was measured for 3 mW (10 mW) laser power as shown in Figs. 3(a) and 3(b). The ratio of photoresponse with laser on to off, for S1-EXF, is ∼3.5; for S2-CVD ∼47 times at 16 V drain voltage. The ratio of photoresponse with laser on to off, for S2-CVD, can be estimated as ∼14, by taking an almost linear dependence of drain current–drain voltage dependence (Fig. 2(b)) between 5 V and 16 V drain voltage into account. The decay and rise times of the photoresponse upon turning the laser on and off can be resolved by 100 ms which is the limitation of the measurement system. The decay and rise times can be determined by fitting the decaying and rising parts of the data with a single exponential function. The photoresponse of S1-EXF which is two orders of magnitude higher than that of S2-CVD, shows a decay and rise time of 6 s and 3 s, respectively. This is much longer timescale compared to only 100 ms for both the decay and the rise times for S2-CVD. The data for S2-CVD are limited by the system and it could be actually much less than 100 ms.

In fact, the trap states due to the defect centers are the main reason for the slower photoresponse in n-type S1-EXF. The cartoon on the upper (lower) panel of Fig. 3(a) describes the movement of

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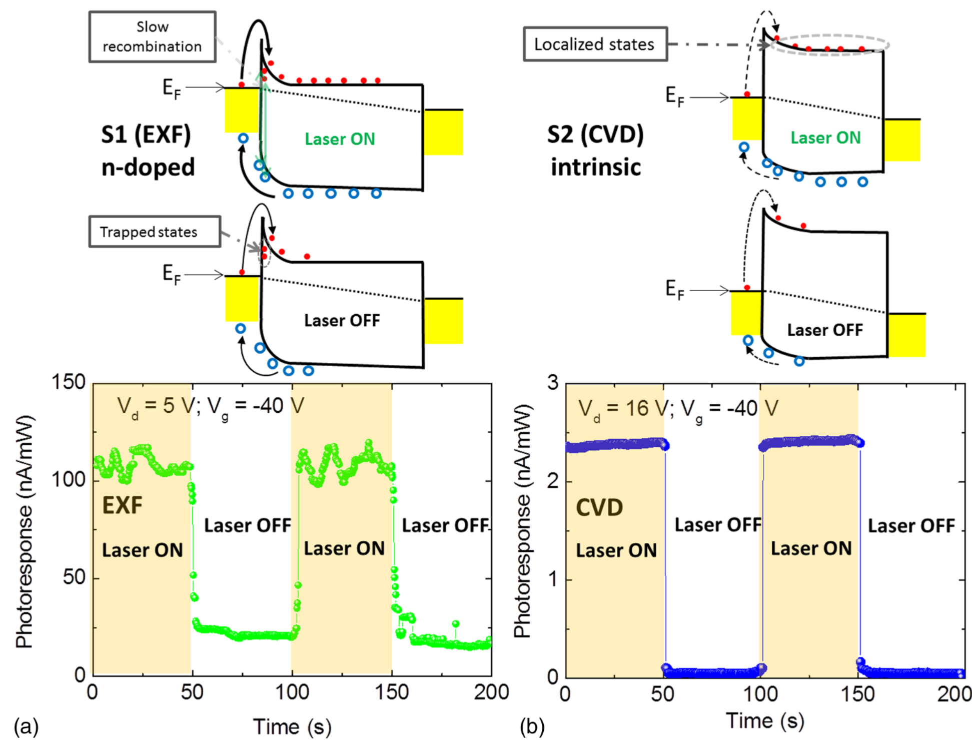

FIG. 3. Time-resolved photoresponse data with laser on and off are shown in (a) for S1-EXF with laser power 3 mW and (b) for S2-CVD with laser power 10 mW. Both decay and rise in photoresponse can be fitted by single exponential. For S1-EXF, the decaying and rising times are 6 s and 3 s, respectively. For S2-CVD, both time scales are 100 ms. Cartoons, in the panels above, describe the simple band diagrams in both samples when laser is on and off. The barrier is shown only for the contact where the drain voltage is applied. Red full (blue open) circles represent electrons (holes). The Fermi level is E_F, the systems are at −40 V gate voltage (V_g) and 5 V drain voltage (V_d) for S1-EXF, 16 V (V_d) for S2-CVD. The overall band of WS_2 FET with respect to electrodes moves slightly down when the laser is on; it moves upward when the laser is off, accompanied by decrease in the photoinduced carriers. There are trapped states due to the defects and related n-type doping at the barrier, in the case of S1-EXF. Charge carriers are mostly in localized states and electrical transport is via photoconduction in S2-CVD, since it is intrinsic.

charges for laser illumination on (off), for a drain voltage of 5 V and −40 V gate voltage. When the laser is off, some electrons are trapped in S1-EXF (lower panel of Fig. 3(a)). Upon turning the laser on, electron-holes are formed and recombine, while the trapped charges are delayed for recombination. Therefore, the decay time of the photoresponse gets longer until the trapped charges are liberated as in the upper panel of Fig. 3(a). Once the laser is back on, first the trapped states will be filled before photo-induced carriers contribute to the current. On the other hand, in S2-CVD, defects can be assumed to be negligible, based on the photoresponse results. The upper (lower) panel of Fig. 3(b) pictures the situation for an intrinsic 2D semiconductor at −40 V gate voltage and 16 V drain voltage, for laser on (off). Charge carriers are mostly in localized states and electrical transport takes place via photoconductive states,[32] as described in the upper panel of Fig. 3(b). Once the laser is off, the transport will be limited by thermally activated process, as in the lower panel of Fig. 3(b).

Based on these results, monolayer CVD grown WS_2 can be confidently pronounced as a visible range, fast photoactive material and photodetector. Although photoresponsivity is fairly low for this device, there is room for improvement. Timescales are expected to be much smaller than 5 ms measured for the multilayer WS_2.[28] Very recently, in a new 2D semiconductor multilayer phosphorene, a response time of 1 ms was measured.[33] However, this material is quite unstable and not robust to environment, which makes it not suitable for applications yet. Rather, stable and large area CVD WS_2 is more promising for future photoactive devices, such as p-i-n photodiodes and photomultipliers.

In summary, we studied low temperature photoresponse of a monolayer exfoliated WS_2 FET device which exhibits n-type behaviour, large dark current, and low tunability in off state by laser

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illumination; while the monolayer CVD WS_2 FET device exhibits intrinsic semiconductor behaviour, small dark current, and high tunability in off state by laser illumination. Time resolved measurements of illumination reveal that the ratio of photoresponse with laser on to off is about four times better in the CVD device and response times are as short as the measurement limit of 100 ms which is much faster than that of the exfoliated one (few seconds). These results support the earlier measurements claiming that there are relatively low native defects in CVD grown WS_2[18] and the fast photoresponse makes CVD WS_2 a suitable optoelectronic material for photoactive devices.

This work was supported in part by MOE Tier1 MOE2013–T1–2–235, MOE Tier 2 MOE2012-T2–2–049, National Research Foundation under NRF RF Award No. NRFRF2010–07, and A*Star SERC PSF grant 1321202101.

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